ARTICLE VI.

On the Perchlorate of the Oxide of Ethule or Perchloric Ether. By Clark Hare and Martin H. Boyè. Read December 4, 1840.

THE energetic properties of perchloric acid, and its stability, compared with the other compounds of chlorine with oxygen, led us to the belief that this acid might be combined with the substance which performs the part of a base in that class of organic salts which are generally designated by the name of *ethers*, and for which Berzelius, in consequence of his theoretical views, has adopted the name of oxide of ethule. For this purpose a concentrated solution of perchlorate and sulphovinate of barytes, in equivalent proportions, was subjected to distillation. The sulphovinate of barytes may be considered as a double sulphate of barytes and the oxide of ethule; and we anticipated that, when heat was applied, a double decomposition would take place between the latter and the perchlorate of barytes. So long as the salts remained in solution no reaction occurred, but as soon as they became solid in consequence of the distillation of the water, a reciprocal decomposition ensued, and a sweet ethereal liquid distilled into the receiver. This *liquid* is the *perchlorate of the oxide of ethule.*

As this substance is extremely explosive, in order to prepare it with safety it is necessary to operate on small quantities. We have employed from seventy to ninety grains of crystallized sulphovinate of barytes, with an equivalent proportion of perchlorate of barytes*; but we would recommend, especially on the first

* The amount of barytes in the perchlorate should be ascertained by an experiment, as it retains water with great tenacity. It may be worth while to mention, that the perchlorate of potassa can-

performance of the experiment, the employment of considerably smaller quantities. The salts should be intimately mixed in a mortar, and placed in a small retort attached to a refrigerator containing ice, and a receiver similarly cooled. The retort is to be heated in an oilbath, in which a thermometer is suspended, so as to indicate the temperature. A wooden screen, furnished with openings covered with thick plate-glass at such intervals as to afford a full view of the different parts of the apparatus, should be erected in front of it, and strings passed around the screen and attached to a bar traversing on a pivot, and supporting an argand spirit lamp, by which heat is communicated to the oilbath, so as to enable the flame of the lamp to be removed from or applied to the apparatus, according to the indications of the thermometer, without exposing the person of the operator. After the heat has reached 212° F., below which the salts employed do not react on each other, it should be raised very gradually, and the distillation finished below 340° F. Under these circumstances but little danger is to be apprehended from the retort, but the ether in the receiver must be treated with the greatest caution, since it has exploded in our hands in attempting to remove it with a pipette from the stratum of water which covers it. This water, therefore, should be removed by the cautious use of strips of blotting paper, moistened at the end, and introduced into the tube employed as a receiver.

To avoid the danger attendant on the management of the ether in its pure state, it may be received in strong alcohol, since it is not explosive when dissolved in alcohol. If the experiment be performed with seventy grains of sulphate of barytes, from one to two drachms of absolute alcohol will be found sufficient for this purpose. By the addition of an equal volume of water, the ether may subsequently be separated from this solution, in small quantities, for the purpose of examination. But, in this case a loss of ether is sustained, by the decomposing influence of the water employed.

The perchlorate of ethule obtained in this way is a transparent, colourless liquid, possessing a peculiar, though agreeable smell, and a very sweet taste, which, on subsiding, leaves a biting impression on the tongue, resembling that of the oil of cinnamon. It is heavier than water, through which it rapidly

not be substituted for the perchlorate of barytes, since the sulphovinate is decomposed without acting on it. We were equally unsuccessful in an attempt to procure the ether by the distillation of perchlorate of barytes and concentrated sulphovinic acid.

sinks. It explodes by ignition, friction, or percussion, and sometimes without any assignable cause. Its explosive properties may be shown, with but little danger, by pouring a small portion of the alcoholic solution into a small porcelain capsule, and adding an equal volume of water. The ether will collect in a drop at the bottom, and may be subsequently separated by pouring off the greater part of the water, and throwing the rest on a moistened filter, supported by a wire. After the water has drained off, the drop of ether remaining at the bottom of the filter may be exploded either by approaching it to an ignited body, or by the blow of a hammer. We are induced to believe that, in explosive violence, it is not surpassed by any substance known in chemistry. By the explosion of the smallest drop, an open porcelain plate will be broken into fragments, and by that of a larger quantity, be reduced to powder. In consequence of the force with which it projects the minute fragments of any containing vessel in which it explodes, it is necessary that the operator should wear gloves, and a close mask, furnished with thick glass-plates at the apertures for the eyes, and perform his manipulations with the intervention of a moveable wooden screen.*

In common with other ethers, the perchlorate of ethule is insoluble in water, but soluble in alcohol; and its solution in the latter, when sufficiently dilute, burns entirely away without explosion. It may be kept for a length of time unchanged, even when in contact with water; but the addition of this fluid, when employed to precipitate it from its alcoholic solution, causes it partially to be decomposed. Potassa, dissolved in alcohol, and added to the alcoholic solution, produces, immediately, an abundant precipitate of the perchlorate of that base, and, when added in sufficient quantity, decomposes the ether entirely. It would appear, therefore, impracticable to form either perchlorovinates or perchlorovinic acid.

We have subjected the perchlorate of ethule to the heat of boiling water without explosion or ebullition.

It may be observed that this is the first ether formed by the combination of an inorganic acid containing more than three atoms of oxygen with the oxide of ethule, and that the chlorine and oxygen in the whole compound are just sufficient to form chlorohydric acid, water and carbonic oxide with the hydrogen and carbon.

* Having suffered severely on several occasions from the unexpected explosion of this substance, we would earnestly recommend the operator not to neglect the precautions mentioned above.

The existence of a compound of the oxide of ethule with an acid containing *seven* atoms of oxygen led us to attempt to combine, by the same method, this base with nitric acid. For this purpose we subjected a mixture of sulphovinate and nitrate of barytes to the same treatment as described above, but the reaction, even when conducted with the greatest possible care, is destructive, hyponitrous ether and gaseous matters being the principal products obtained. Nor were we more successful in our attempts to procure a sulphurous or hyposulphuric ether by the same process.